# (19) World Intellectual Property Organization International Bureau



# 

# (43) Internati nal Publication Date 8 November 2001 (08.11.2001)

# **PCT**

# (10) International Publication Number WO 01/83654 A1

(51) International Patent Classification<sup>7</sup>: C11B 1/04, 1/06, A23D 9/00, 7/00, A23L 1/24, 1/39

(21) International Application Number: PCT/EP01/04711

(22) International Filing Date: 25 April 2001 (25.04.2001)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data: 00303746.2 4 May 2000 (04.05.2000) E

(71) Applicant (for all designated States except AG, AU, BB, BZ, CA, CY, GB, GD, GH, GM, IE, IL, IN, KE, LK, LS, MN, MW, MZ, NZ, SD, SG, SL, SZ, TT, TZ, UG, ZA, ZW): UNILEVER N.V. [NL/NL]; Weena 455, NL-3013 AL Rotterdam (NL).

(71) Applicant (for AG, AU, BB, BZ, CA, CY, GB, GD, GH, GM, IE, IL, KE, LK, LS, MN, MW, MZ, NZ, SD, SG, SL, SZ, TT, TZ, UG, ZA, ZW only): UNILEVER PLC [GB/GB]; Unilever House, Blackfriars, London, Greater London EC4P 4BQ (GB).

(71) Applicant (for IN only): HINDUSTAN LEVER LTD [IN/IN]; Hindustan Lever House, 165-166 Backbay Reclamation, 400 020 Mumbai (IN).

(72) Inventors: MAITLAND, Reinout; Unilever Patent Department, Olivier van Noortlaan 120, NL-3133 AT Vlaardingen (NL). PUTTE, Karel, Petrus, Agnes, Maria, Van; Unilever Patent Department, Olivier van Noortlaan 120, NL-3133 AT Vlaardingen (NL). STAVRIDIS, Vangelis; Elais S.A., 74 Pireos Street, GR-185 47 n Faliro (GR). TORNALETTI, Nicola; VAN DEN BERGH ITALY SPA, Div. Dev. MGR, Corso Europa 24, Inveruno, I-Milano (IT).

(74) Agent: SIKKEN, Antonius, H., J., M.; Unilever Patent Department, Olivier van Noortlaan 120, NL-3133 AT Vlaardingen (NL).

(81) Designated States (national): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, UZ, VN, YU, ZA, ZW.

(84) Designated States (regional): ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

#### Published:

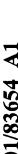
with international search report

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

(54) Title: METHOD FOR PROCESSING OLIVES

(57) Abstract: It has been found that the level of polyphenols in an olive oil can be increased by adding water to the olives during the crushing of the olives. In particular the invention concerns a method for processing olives obtaining an olive oil, comprising the steps of a) crushing the olive through a sieve in the presence of water in an amount of 1 to 40% by weight, based on the weight of the olives; b) malaxation of the olives obtained in step a); c) decanting the olive oil.





1

# Method for processing olives

5

The present invention relates to a method for processing olives for obtaining an olive oil with an increased amount of polyphenols.

10 Olive oil is a product which is of increasing interest, not only for its taste but also for the possible health improving properties. Some of these health improving properties are attributed to the presence of so-called minor nutrients in the olive oil, in particular anti-oxidants such as polyphenols. It 15 has recently been found that such anti-oxidants positively interfere with the body's cardiovascular system.

The present invention is directed towards increasing the amount of polyphenols in the olive oil. Polyphenols are known to be 20 anti-oxidants which are present in olive fruits. Usually polyphenols are present not as a single compound but as a mixture of different polyphenols. Polyphenols are compounds which share a phenolic hydroxyl group. Olive fruit originating polyphenols comprise oleuropein, aglycons, tyrosol and 25 hydroxytyrosol.

The object of the present invention is to increase the level of polyphenols in olive oil, by means of a relative simple method. The object is further that such a simple method is applicable in conventional processes that are used for processing olives without any extra costs. Minimal additional equipment is necessary.

2

In general, the conventional process for processing olives comprises the following steps:

- 1. harvesting the olive fruits, removal of any leaves and washing
- 5 of the olive fruits;
  - 2. crushing;
  - 3. malaxation;
  - 4. decanting (by centrifugation) of the olive oil;
  - 5. further centrifugation.

10

Different variations of this process are known. For instance in FR 2,195,677 it is described that the olives are not crushed or milled but are transferred directly to a first malaxation apparatus. After processing in the first malaxation apparatus

- 15 the malaxation mass obtained is transferred to a second malaxation apparatus.
  - In GB 741,422 a process is described for increasing the amount of oil recovered from cellular vegetable materials such as
- 20 olives. A liquid medium, such as water, in an amount of 3 to 30 times the weight of the fat containing material, delivers a high speed series of impacts, rupturing the oil or fat containing cells. The particle size of the solid residues obtained is larger than 50 μm. After the step of rupturing the cells, the
- 25 mixture obtained is separated into a fat phase and a liquid and/or solids containing phase.

According to the invention it has been found that the level of polyphenols in an olive oil can be increased by adding water to 30 the olives during the crushing of the olives. It has been shown that the addition of water causes fine fruit particles, which are hydrophilic, to be entrained in the added water, and

WO 01/83654

thereafter in the produced olive oil. This effect will contribute to the polyphenols content of the olive oil.

Therefore, according to the invention a method is provided for 5 processing olives obtaining an olive oil, comprising the steps of

- a) crushing the olives through a sieve;
- b) malaxation of the olive paste obtained in step a);
- c) decanting the olive oil;
- 10 characterized in that

in step a) water is added in an amount of 1 to 40 % by weight based on the weight of the olives.

During step a) of the process olive oil fruits are crushed,

15 preferably in a hammer mill equipped with a perforated sieve.

By means of crushing oil is released from the plant tissue. The hammers rotate inside the sieve with a tip speed that is for example 65 m/sec. Conventionally the sieve will have a mesh of 6 to 8 mm. The present inventors have found that the

- 20 polyphenols content can be further increased by using a sieve having a mesh of 1 to 6 mm, preferably 1 to 4 mm. By using a smaller sieve the fraction of smallest fruit particles will be maximized.
- 25 The amount of water added is within the ranges indicated above. However, depending on the ripeness of the olive fruit, the amount of water added can be varied. The ripeness of the olive fruit is determined by means of the ripeness index, as described for instance by Ranalli and Morelli, Leatherhead Food 30 RA Food Industry Journal, Vol. 2, winter 1999.

4

The ripeness index defines the maturity of the olive as a function of fruit color in both skin and pulp. Samples of olives, 100 for each variety, are taken at random and are classified into the following categories: 0 = olives with intense green or dark green epidermis (a); 1 = olives with yellow or yellow-green epidermis (b); 2 = olives with reddish yellow epidermis (c); 3 = olives with reddish or light violet epidermis (d); 4 = olives with black epidermis, and totally white pulp (e); 5 = olives with black epidermis, and violet 10 pulp to the midpoint (f); 6 = olives with black epidermis, and violet pulp almost to the pit (g); 7 = olives with black epidermis, and totally dark pulp (h). The ripeness index is calculated from equation 1, where alphabetic variables indicate the number of fruits in each category.

15

$$RI = (a \times 0 + b \times 1 + c \times 2 + d \times 3 + e \times 4 + f \times 5 + g \times 6 + h \times 7)/100$$
 [1]

Ripeness values are between 0 and 7.

20 It has been found that the higher the ripeness index, the less water will need to be added to the olives. Therefor, for olives having a ripeness index of 0 to 4, the water added is preferably 13 to 28 %. For olives having a ripeness index of larger than 4, the water added is preferably 1 to 12 % by weight.

It was further found that the crusher throughput is a factor that also influences the amount of polyphenols in the olive oil obtained. This optimum is influenced by the type and maturity 30 of the olives, as described above, and also by the choice of the mesh of the sieve. The residence time of the olives in the crusher is from 2 seconds to 5 minutes, preferably 2 to 20 seconds.

5

According to a further aspect of the invention, the water added in step a) contains citric acid as a processing aid. The citric acid will increase the amount of polyphenols in the final olive 5 oil. A further advantage of the use of citric acid is that it has a protective function by chelating metals, which prevents the metals of being catalysts for the oxidation of the polyphenols. The water preferably contains 0.5 to 7 % by weight citric acid, based on the weight of the water.

10

Preferably as the source of citric acid lemon juice is added.

In general lemon juice contains about 7 % by weight citric acid. The lemon juice can be added as such to the water, as long as the citric acid content is within the ranges indicated 15 above. It is also possible to add whole lemons or lemon parts to the water.

In step b) malaxation of the olive paste obtained in step a) is carried out. Malaxation serves to favor the separation of the 20 liquid phases from solids and to break up oil-water emulsions. It facilitates the coalescence of oil droplets and thereby directly influences the oil yield. After crushing up to 45 % of the oil droplets will have a particle size larger than 30 µm whereas after malaxation more than 80 % of the oil droplets 25 will have a particle size larger than 30 µm. Malaxation can be carried out from 10 to 90 minutes. It can proceed under conventional process conditions, for instance at 30 °C and 30 rpm for 30 minutes.

30 In step c) olive oil is separated from solid phase and aqueous phase. In this step only the coarsest solid matter is removed. The fines, with a high polyphenols content, should be retained in the oil, preferably substantially remaining in the aqueous

6

phase dispersed in the oil. This step can be carried out by means of a centrifuge. Preferably, the decanting step is carried out such that the resulting olive oil has a residual water content of less than 3 wt.%, preferably 0.5 to 1 wt.%.

5 When citric acid has been added to the water, the residual citric acid content in the olive oil will be from 5 ppm to 5000 ppm.

The polyphenols content can be optimized by means of the 10 throughput through the decanter. In general a higher throughput will result in separation of only the heaviest fraction and therefore more olive particles will remain in the olive oil.

After the decanting step an olive oil will be obtained which
15 still contain some olive particles. The solids content of the
olive oil, based on the weight of the oil, after decanting is
preferably at least 0.05 wt. %. The upper limit for the solids
content is preferably 5 wt. %, more preferably 1 wt. %. Solids
content can for instance be measured by passing the olive oil
20 obtained through a sieve having a mesh of 0.01 µm and weighing
the residue. The average particle size of the olive particles
is less than 50 µm, preferably less than 20 µm, more preferably
less than 5 µm. The lower limit for the particle size is 0.01
µm in order to characterize the particles as solids. Average
25 particle size can be determined by methods known in the art.

For some applications it can be desirable to have a clear oil.

In that case the olive oil can be purified further to remove at least part of the remaining olive particles, for instance by

30 means of centrifugation. It has been found that even in that case an olive oil will be obtained that has a higher

7

polyphenols content than those obtained with conventional methods.

The present invention also relates to the olive oil obtained 5 with the above described method. Generally, the olive oil of the invention has a polyphenols content higher than 300 ppm. In case of the processing of unripe olives having a ripeness index of 0 to 4, the polyphenols content is higher than 500, preferably higher than 1000 ppm. In case of the processing of 10 ripe olives having a ripeness index of more than 4, the polyphenols content is higher than 300 ppm, preferably higher than 400 ppm. The oil obtained has a nice taste with minimal bitterness or astringency as compared to conventional olive oils having a high polyphenols content.

15

The total content of polyphenols in olive oils can be established by standard methods, e.g. by the colorimetric Gutfinger method as described in J.Am.Oil.Chem.Soc. 1981, 11, pp. 966-968, which method is based on the reaction of a 20 methanolic extract of olive oil and the Folin-Ciocalteau reagent. This method has been used for establishing the polyphenols values mentioned in the present patent specification.

25 The olive oil of the invention can be used in the preparation of food products such as a spread, salad dressing, mayonnaise or sauce. Spreads are food compositions which usually contain a substantial amount of fat, often 40 wt.% or more. Usually the fat consists of a liquid oil and a structuring fat which gives 30 the fat blend a proper consistency. Sauces are meant to include any type of sauce, for instance sauces that are ready to use, in particularly after having been heated, such as for instance

8

tomato sauces. Processes for the manufacture of these products are well known in the art and need no illustration.

The olive oil can also be blended with edible oils, such as

5 vegetable oils, examples of which are rapeseed oil, sunflower
seed oil, soybean oil and corn oil. The invention is not limited
to oils which are devoid of any polyphenols, either by nature or
because of a refining process, but also of oils which contain
polyphenols of their own such as virgin olive oils. Examples of

10 other olive oils which can be mixed with the oil according to
the present invention are an extra virgin olive oil, a fine
virgin olive oil, a semi-fine or regular virgin olive oil, a
refined olive oil or an olive residue oil but also an olive oil
blend, which contains part virgin olive oil and part refined

15 olive oil.

According to a second embodiment of the present invention a method for preparing an olive paste is provided comprising the steps of

- 20 a) crushing olives through a sieve, wherein water is added in an amount of 1 to 40 % by weight, preferably 12 to 28 % by weight based on the weight of the olives;
  - b) malaxation of the olives;
  - c) separating olive kernel particles from the paste.
- 25 The preferred crushing and malaxation conditions are the same as described above.

The olive paste obtained with this process has a high level of polyphenols and is suitable for food applications, in

30 particular in food products having a fat phase. Said paste can be used in food products, such as olive oils, spreads, mayonnaise, sauces, etc. to replace the fat phase normally used in the production of these products.

9

## Example 1

An industrial milling was applied, with a hammer crusher equipped with a 4mm mesh sieve. 430 kg olives (Tsounati 5 variety) were crushed in this mill while adding 70.5kg water (16.4 wt.%). Malaxation of the resulting paste was performed at 30 °C and 30 rpm for 30 minutes. The decanter was operated at such a speed that the residual water content of the oil was 3.2 wt.%. The obtained oil is characterized by the fact that it 10 contains 3.2 wt.% water, 0.54 wt.% olive particles with an average particle size of 11.0 µm and 1100 ppm polyphenols. The taste of the product presented minimal bitterness and astringency.

# 15 Example 2

The oil from Example 1 is further processed as if a conventional process scheme was applied. The oil is centrifuged in a standard vertical centrifuge. The obtained reference oil is characterized by the fact that it contains only 0.28 wt.% 20 water, 0.03 wt.% olive particles and 269 ppm polyphenols.

# Example 3

In the same mill mentioned in Example 1 414kg olives (Tsounati variety) were crushed while adding 118.4kg water (28.6 wt.%).

25 Malaxation of the resulting paste was performed at 30 °C and 30 rpm for 30 minutes. The decanter was operated at such a speed that the residual water content of the oil was 1.3 wt.%. The obtained oil is characterized by the fact that it contains 1.3 wt.% water, 0.32 wt.% olive particles with an average particle 30 size of 13.6 μm and 868 ppm polyphenols. The taste of the

product presented minimal bitterness and astringency.

10

# Example 4

In a lab scale hammer mill, equipped with a 2 mm mesh sieve, 100 g olives (Koroneiki variety) were crushed while adding 30 g water (30 wt.%) containing 1.44 g citric acid (4.8 wt.%).

5 Malaxation of the resulting paste was performed at 30 °C for 30 minutes. After centrifugation of the paste, the obtained oil is characterized by the fact that it contains 960 ppm polyphenols and 2434 ppm citric acid. The presence of citric acid in the oil was not detected by tasting.

11

#### Claims

- A method for processing olives obtaining an olive oil, comprising the steps of
- a) crushing the olives through a sieve;
- b) malaxation of the olives obtained in step a);
- c) decanting the olive oil; wherein in step a) water is added in an amount of 1 to 40 % by weight, based on the weight of the olives.
- 2. A method according to claim 1, wherein the olives are crushed through a sieve having a mesh of 1 to 6 mm.
- 3. A method according to claim 1, wherein the olives are crushed through a sieve having a mesh of 1 to 4 mm.
- 4. A method according to claim 1, wherein the decanting step is carried out such that the resulting olive oil has a residual water content of less than 3 wt.%.
- 5. A method according to claim 1, wherein the decanting step is carried out such that the resulting olive oil has a residual water content of 0.5 to 1 wt.%.
- 6. A method according to claim 1, which further includes removing at least part of the olive particles remaining in the olive oil.
- 7. A method according to claim 1, wherein olives having a ripeness index of 0 to 4 are processed and wherein water is added in an amount of 13 to 28 % by weight.

12

- 8. A method according to claim 1, wherein olives having a ripeness index of more than 4 are processed and wherein water is added in an amount of 1 to 12 % by weight.
- 9. A method according to claim 1, wherein the water added in step a) contains citric acid.
- 10. A method according to claim 1, wherein the water added in step a) contains 0.5 to 7 % by weight of citric acid, based on the weight of the water.
- 11. A method according to claim 9, wherein lemon juice is used as the source of the citric acid.
- 12. An olive oil obtained with the method of claim 7, having a polyphenols content higher than 500 ppm and a solids content after decanting of at least 0.05 wt.%.
- 13. An olive oil obtained with the method of claim 7, having a polyphenols content higher than 1000 ppm and a solids content after decanting of at least 0.05 wt.%.
- 14. An olive oil obtained with the method of claim 8, having a polyphenols content higher than 300 ppm and a solids content after decanting of at least 0.05 wt.%.
- 15. An olive oil obtained with the method of claim 8, having a polyphenols content higher than 400 ppm and a solids content after decanting of at least 0.05 wt.%.

13

- 16. An olive oil according to claim 12 or 13 and having a solids content of at most 5 wt.%.
- 17. An olive oil according to claim 14 or 15 and having a solids content of at most 5 wt.%.
- 18. An olive oil according to claim 16 or 17 containing olive particles having a particle size smaller than 50 μm.
- 19. An olive oil according to claim 16 or 17 containing olive particles having a particle size smaller than 20 μm.
- 20. An olive oil according to claim 16 or 17 containing olive particles having a particle size smaller than 5 µm.
- 21. A food product containing an olive oil according to claim 13 or 15.
- 22. A food product according to claim 21, which is a vegetable oil, preferably an extra virgin olive oil.
- 23. A food product according to claim 21, which is a spread, mayonnaise, salad dressing or tomato sauce.
- 24. A method for preparing an olive paste, comprising the steps of
- a) crushing olives through a sieve, wherein water is added in an amount of 1 to 40 % by weight based on the weight of the olives;
- b) malaxation of the olives;
- c) separating olive kernel particles from the paste.

14

- 25. A method for preparing an olive paste, comprising the steps of
- a) crushing olives through a sieve, wherein water is added in an amount of 12 to 28 % by weight, based on the weight of the olives;
- b) malaxation of the olives;
- c) separating olive kernel particles from the paste.
- 26. An olive paste obtained by the process of claim 24.
- 27. A food product containing an olive paste according to claim 26.

### INTERNATIONAL SEARCH REPORT

Int onal Application No PCT/EP 01/04711

A. CLASSIFICATION OF SUBJECT MATTER IPC 7 C11B1/04 C11B A23D7/00 C11B1/06 A23D9/00 A23L1/24 A23L1/39 According to International Patent Classification (IPC) or to both national classification and IPC B. FIELDS SEARCHED Minimum documentation searched (classification system followed by classification symbols) IPC 7 C11B A23D A23L Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched Electronic data base consulted during the international search (name of data base and, where practical, search terms used) EPO-Internal, WPI Data, PAJ, FSTA C. DOCUMENTS CONSIDERED TO BE RELEVANT Relevant to claim No. Category \* Citation of document, with indication, where appropriate, of the relevant passages WO 99 52377 A (UNILEVER PLC ;UNILEVER NV 9,10, X 13-15 (NL)) 21 October 1999 (1999-10-21) page 4, line 30 -page 5, line 7 example 1 claims 1-3,9,10 EP 0 849 353 A (UNILEVER PLC ;UNILEVER NV 9,10. X 13-15 (NL)) 24 June 1998 (1998-06-24) page 2, line 8 - line 9 claim 1 GB 741 422 A (BRITISH GLUES AND CHEMICALS 1 A LIMITED) 7 December 1955 (1955-12-07) cited in the application example 5 claims 7,8,15-19 -/--X Further documents are listed in the continuation of box C. Patent family members are listed in annex. Special categories of cited documents: "T" later document published after the international filing date or priority date and not in conflict with the application but clied to understand the principle or theory underlying the "A" document defining the general state of the art which is not considered to be of particular relevance invention "E" earlier document but published on or after the international filing date "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified) "V" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art. "O" document referring to an oral disclosure, use, exhibition or \*P\* document published prior to the international filing date but later than the priority date claimed "&" document member of the same patent family Date of the actual completion of the international search Date of mailing of the international search report 27/07/2001 20 July 2001 Name and mailing address of the ISA Authorized officer European Patent Office, P.B. 5818 Patentlaan 2 NL – 2280 HV Rijswijk Tel. (+31–70) 340–2040, Tx. 31 651 epo nl, Fax: (+31–70) 340–3016 Dekeirel. M

# INTERNATIONAL SEARCH REPORT

in onal Application No PCI/EP 01/04711

		101/21 01/04/11					
C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT  Category Citation of document, with Indication, where appropriate, of the relevant passages  Retevant to claim No.							
odichola ,	Common or provincest was averaged, waste abbrohimes or mis tessage beorges		I MAGRETT TO CHARLES 140.				
A	FR 2 195 677 A (PAGES HENRI) 8 March 1974 (1974-03-08) cited in the application page 2, line 6 - line 8 page 3, line 9 - line 11 figure 3		1				
A	DATABASE WPI Section Ch, Week 198744 Derwent Publications Ltd., London, GB; Class D23, AN 1987-308641 XP002149642 -& ES 8 707 289 A (BERGES L C M), 1 October 1987 (1987-10-01) abstract		1				
	<del></del>	•					
			i				
!							
		•					
	•						
		·					
			•				
		*					
		•					

# INTERNATIONAL SEARCH REPORT information on patent family members

onal Application No PCT/EP 01/04711

Patent document cited in search report		Publication	Patent family	Publication date
		date	member(s)	
WO 9952377	A	21-10-1999	AU 733005 B	03-05-2001
			AU 3598599 A	01-11-1999
			EP 1071338 A	31-01-2001
			TR 200002967 T	22-01-2001
			US 6162480 A	19-12-2000
EP 0849353	A	24-06-1998	US 5998641 A	07-12-1999
			DE 69704330 D	26-04-2001
			ES 2155653 T	16-05-2001
			TR 9701662 A	21-09-1999
GB 741422	А	07-12-1955	NONE	
FR 2195677	A	08-03-1974	NONE	
ES 8707289	Α	01-10-1987	ES 556119 D	16-07-1987